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WORKSHOPS ON THE FEDERAL REFERENCE METHOD FOR DETERMINATION OF INHALABLE PARTICLES (1979, 1980)

by

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The Environmental Protection Agency (EPA) is considering promulgating a new ambient air quality standard for inhalable particles (IP). An immediate consequence of the new standard will be the specification of a federal reference method for measuring inhalable particles. Two workshops concerned with this subject were sponsored by EPA's Environmental Monitoring Systems Laboratory. The first workshop was held at Research Triangle Park, North Carolina on October 1 and 2, 1980. At this workshop recent developments in particle sampling were presented. Tentative test procedures for demonstrating sampler performance were also discussed. The second workshop was held at Fort Collins, Colorado on November 18-20, 1980. Testing procedures and wind tunnel requirements were formulated and/or revised during this workshop.

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PREFACE

The Environmental Protection Agency (EPA) is considering promulgating a new ambient air quality standard for inhalable particles (IP). An immediate consequence of the new standard will be the specification of a Federal reference method for measuring inhalable particles. Two workshops concerned with this subject were sponsored by the Environmental Monitoring Systems Laboratory (EMSL) of EPA: The first workshop was held at Research Triangle Park, North Carolina, on October 1 and 2, 1979, and the second, at Fort Collins, Colorado, November 18-20, 1980. Part I of this report summarizes the 1979 workshop presentations; Part II summarizes the 1980 workshop. Section 2 of each part presents a synopsis of the respective workshop for those persons who do not wish to read the entire summary.

Some explanation of the procedures used to prepare these summaries is in order. For the 1979 IP Workshop, R. P. Donovan of Research Triangle Institute took detailed notes, which provided a basis for the summary. Tape recordings of this workshop served as a verification of his notes. For better clarity, the order in which the presentations and discussions appear has been rearranged and does not represent the order in which they occurred. The pooled notes of E. R. Kashdan and M. B. Ranade (Research Triangle Institute) and L. J. Purdue and K. A. Rehme (EPA/EMSL) were the basis for the summary of the 1980 workshop. A draft summary was prepared soon after each workshop and sent to attendees for review. Reviewer comments helped to eliminate inaccuracies and misstatements. Footnotes have been used liberally to both cross-reference discussions of key topics at the different workshops and to identify literature cited during presentations.

Special thanks are extended to Dr. Walter John (Department of Health Services, State of California) for his prompt and conscientious review of both summaries. Thanks are also extended to Dr. Billy Loo (Lawrence Berkeley Laboratories) and Dr. Bruce Appel (Department of Health Services, State of California) for providing copies of the material they presented at the 1980 workshop.

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PART I

SUMMARY OF THE OCTOBER 1979 WORKSHOP ON THE FEDERAL REFERENCE METHOD FOR DETERMINATION OF INHALABLE PARTICLES

SECTION 1

INTRODUCTION

The current national ambient air quality standard (NAAQS) for particulate matter is the total suspended particles (TSP) standard which was established in 1971 by the Environmental Protection Agency (EPA). It has been recognized for a long time that measurements of TSP using the high-volume (hi-vol) sampler may not provide the best measure of population exposure because very coarse particles can be collected by the hi-vol which probably represent a minimal health hazard. The Clean Air Act amendments of 1977 require EPA to review the scientific criteria upon which the particulate matter standard is based. A possible and reasonable response to this review would be the establishment of a size-specific standard for particulate matter.

Miller et al. (1979)* reviewed a number of considerations for establishing a size-specific particle standard. Their pertinent recommendations are: (1) that mouth breathing should be addressed by the standard, which implies that larger particles need be considered since mouth breathing permits greater deposition in the conducting airways and gas-exchange areas than nose breathing; (2) that 15 μm be used as the cutpoint for defining inhalable particles (IP); and (3) that a second cutpoint of 2.5 μm be used to measure particles having predominant penetration to the gas-exchange region of the respiratory tract.

In addition to the difficult tasks of defining the size of particles in an IP standard and setting the level of the standard, there remains the task of specifying a reference method to determine compliance with the standard. On October 1 and 2, 1979, a workshop concerning the development

*Miller, F. J., et al. "Size Considerations for Establishing a Standard for Inhalable Particles," JAPCA, 29(6):610-615, 1979.

of the Federal reference method (FRM) for determining inhalable particle concentrations in the atmosphere was held in Research Triangle Park, North Carolina. The workshop was organized by Research Triangle Institute (Ranade and Kashdan) under the sponsorship of EPA's Environmental Monitoring Systems Laboratory (Purdue and Rehme). The purpose of this workshop was to bring together a group of EPA personnel, aerosol researchers, and sampler vendors to discuss various topics including general approaches to and ramifications of establishing an IP reference method, evaluation of existing IP hardware and data, and standardizing procedures for testing IP sampler performance. The workshop was successful in identifying problem areas, generating proposed solutions, and, in a few instances, obtaining a consensus of opinion.

SECTION 2

SYNOPSIS OF 1979 IP WORKSHOP

2.1 OBJECTIVES

The objectives of the 1979 IP Workshop were to bring together a group of EPA personnel, aerosol researchers, and vendors of sampling instruments to discuss aspects of establishing a Federal reference method (FRM) for determining inhalable particles in the atmosphere. The workshop was successful in identifying problems in the evaluation of IP samplers in the field and laboratory, in suggesting solutions to some of these problems, and in pointing out problems that could not be resolved without further study.

2.2 REFERENCE METHOD FOR INHALABLE PARTICLES

2.2.1 Two distinct approaches to specifying a reference method for inhalable particles were identified: (1) the design specification approach, whereby one particular sampler is specified as the reference method sampler and all reference method samplers must be fabricated within close tolerances to the specified sampler design; and (2) the performance specification approach, whereby all reference method samplers need only employ a common principle of operation and be subjected to certain performance tests (in a wind tunnel and in the ambient air) before approval.

2.2.2 EPA personnel and aerosol researchers tended to prefer a performance specification approach because of its flexibility. Vendors, however, preferred the simplicity of the design specification approach.

2.3 EVALUATION OF INSTRUMENTS FOR MEASURING INHALABLE PARTICLES

2.3.1 Laboratory evaluations of the dichotomous samplers revealed that particle separation in the bell-shaped inlet was windspeed dependent.

2.3.2 Field testing of the dichotomous sampler in the IP Network showed that reproducibility of side-by-side samplers, especially for the coarse fraction, was poor. The size-selective hi-vol (design cutpoint of 15 μm) showed better precision.

2.3.3 Evaluation of samplers for source level inhalable particles did not appear to be as problematic inasmuch as a cyclone had been developed that cut sharply at 15 μm.

2.4 PERFORMANCE TESTING OF IP SAMPLERS

2.4.1 Wind tunnel tests of the sampler inlet particle separation was considered necessary if the Federal reference method was to be based on performance specifications. Requirements for the wind tunnel included: a top speed of at least 15 mph (24 km/h); sufficient cross section so that the test device did not block more than a small percentage (tentatively, 5 percent) of the tunnel cross-sectional area; and capability of delivering a uniform cloud at the test section. It was generally agreed that static testing of samplers would not be required as part of any reference method testing.

2.4.2 The experimental design for the inlet testing that appealed to most attendees was to test the inlet with liquid particles (nominally, 5 to 25 μm) and one size of large, solid particles. Liquid particles would be used to evaluate sampling effectiveness and wall losses; solid particles would be used to assess particle bounce problems. The vibrating orifice or perhaps spinning disk aerosol generators were suitable for generating liquid particles, but the choice of material and method of dispersal for the solid aerosol remained unresolved.

2.4.3 Acceptance levels for the inlet separation characteristics were specified by a performance envelope defined by lognormal distributions with parameters MMD = 15 ± 2 μm, σ_g = 1.0 - 1.7. A 10-percent error band at the lower size range was added to allow for experimental error. It was generally acknowledged that the laboratory characterization of the sampler inlet should be evaluated in terms of expected performance in ambient air. Collected mass less than 15 μm was the criterion chosen for this evaluation, assuming various input aerosol distributions (e.g., lognormal).

2.4.4 Other parameters considered desirable to test were flow rate stability and sampler reproducibility. Both parameters could be tested in ambient air, but the protocol and acceptance levels were not defined.

SECTION 3

SUMMARY OF THE 1979 IP WORKSHOP

3.1 IP REFERENCE METHOD APPROACHES

In establishing a Federal reference method (FRM) for determining inhalable particles in the atmosphere, two options, both used historically, are available. In the first option, a particular sampler design is specified as the reference method sampler. Details (design specifications) for fabricating the sampler are provided, which, if followed within close tolerance limits, should ensure that the measurements with two reference method samplers agree reasonably well. The approach here, then, is to base the reference method on a single, standardized sampler (a "design specification approach"). The second, less restrictive, option is to specify a principle of operation (measurement principle) that all reference methods must use, but not to prescribe a particular sampler design. Instead, a controlled test atmosphere is generated and tests of the performance of candidate reference method samplers are made; those samplers that pass the tests within some tolerance limits qualify as reference method samplers. (Here, a "performance specification approach" is taken.) Both approaches have been taken in establishing reference methods for EPA criteria pollutants. For example, total suspended particulate (TSP) matter is, in effect, that which the high-volume sampler measures because a design specification approach was taken. On the other hand, chemiluminescence is the reference measurement principle for ozone, and seven different designs of ozone meters that use this principle have been designated as reference methods.

In his opening remarks to the workshop group, Dr. Thomas Hauser (Director EPA/EMSL) called for assistance from all parties (researchers, vendors, contractors, etc.) in developing the reference method for inhalable particles. Mr. Larry Purdue (EPA/EMSL) then reviewed EPA's approaches to specifying reference methods for criteria pollutants and discussed the relative merits of performance and design specification approaches.

3.1.1 Introductory Remarks, Dr. Thomas Hauser, Director of EPA/EMSL

Summary:

Dr. Hauser gave the opening presentation, which served as an introduction to the workshop. He reviewed Section 109 of the Clean Air Act Amendments of 1977 and cited the provision calling for a review of the national ambient air quality standards (NAAQS) to be completed no later than December 1980. An IP standard is a possible response to this requirement, but it is not clear at present whether it will replace, supplement, or otherwise modify the existing TSP standard. Dr. Hauser preferred to have the best thinking available at the beginning of the Federal reference method development process rather than at the end. EPA's Environmental Monitoring Systems Laboratory (EMSL) has the responsibility for developing the reference method that would be promulgated in support of any inhalable particle standard that the Office of Air Quality Planning and Standards (OAQPS) generates.

Dr. Hauser explicitly stated that the audience included vendors of particulate sampling equipment since input from this segment of the scientific community was felt to be important. He depicted the present meeting as analogous to a 1971 workshop meeting regarding the equivalency regulations for gas analyzers. At that time, vendors asked EPA not to dictate methods but to specify goals.

Conclusions:

No particular discussion followed Dr. Hauser's presentation. It should be noted that the problems confronting EPA in 1971 concerning gas analyzers were different from the problems concerning IP measurement. In the former case, considerable measurement hardware, differing not only in design but also in measurement principle, already existed and hence the need was to establish comparability among the many analyzers. Each vendor was eager to have his particular analyzer designated as an acceptable EPA method and welcomed an approach that permitted flexibility in the design of analyzers. In the latter case, few IP samplers have been developed and they are all fairly similar in both principle and design. Consequently, flexibility in sampler design was not nearly as attractive to the vendors as it was in 1971.

3.1.2 Federal Reference Methods for Criteria Pollutants, Mr. Larry Purdue, EPA/EMSL

Summary:

Mr. Larry Purdue provided additional background information for the workshop. One of the assumptions of the conference was the definition of an inhalable particle as any airborne particle having an aerodynamic diameter of 15 μm or less. In contrast to Dr. Hauser's comments, Mr. Purdue pictured the promulgation of an IP standard as a virtual certainty and repeated the need for this final standard and a Federal reference method by December 1980.

Mr. Purdue then reviewed EPA's past practice concerning Federal reference methods for criteria pollutants. Previous reference methods given in appendixes to the national ambient air quality standards (40 CFR, Part 50) can be divided into two types:

1. Those lending themselves to an automated method (CO, O_3, and NO_2--these reference methods specify a measurement principle and a calibration procedure), and

2. Manual methods (SO_2, TSP, and Pb--these reference methods specify a unique design or procedure).

Supplementing the Part 50 appendixes is a 1975 Part 53 regulation pertaining to ambient air reference and equivalent methods. (Equivalent methods are those that use a measurement principle different than the reference method.) This 1975 Part 53 regulation includes formal requirements and performance specifications for automated reference and equivalent methods. Specific test procedures for designating particular analyzer designs as either reference or equivalent methods are given in these regulations. It also puts the burden of testing upon the manufacturer. This Part 53 regulation is considered a successful regulation, and a general goal at this time is to consider adopting a similar approach for the IP reference method.

The TSP reference method of 1971, specified in Appendix B to Part 50, is a manual method. The high-volume samplers are in effect the only acceptable means to determine compliance for TSP. There is some concern that being locked into this design specification approach for TSP has hampered

the development of new samplers. In contrast to the design specification approach, a performance specification approach was followed for several gaseous criteria pollutants. In the case of O_3, chemiluminescence is the reference measurement principle specified in Part 50. Not only have a number of commercial analyzers been designated reference analyzers under the Part 53 provisions, but an entirely different method based on UV photometry meeting all performance and consistent relationship requirements has been designated as an equivalent method for O_3. (The consistent relationship test is a side-by-side comparison between a reference method and the candidate equivalent method at a field site under ambient conditions.)

Mr. Purdue continued and stated that the first problem was to decide whether to base the IP reference method on performance specifications or on design specifications. An IP reference method based on design specifications would rigidly describe a sampler by using engineering drawings specifying dimensions (with allowable tolerances) for critical areas within the sampler. This approach would restrict reference method samplers to only those that are manufactured in strict conformance with the design specifications prescribed by the Federal reference method. With this approach, candidate equivalent methods would be either those methods based on the same measurement principle as the Federal reference method (filtration and gravimetric determination of mass) but using an alternate sampler design or those methods based on a different measurement principle (e.g., beta gauge determination of mass). A preference now exists within EPA to promulgate an IP Federal reference method based on performance specifications. In this approach, a reference method sampler would be any sampler using the specified measurement principle, provided the sampler meets the prescribed performance specifications. A vendor of a candidate reference method sampler would conduct prescribed tests to demonstrate that the sampler meets the performance specifications, but the design of the sampler would be left in the vendor's hands. Candidate equivalent methods would then be only those samplers using a different measurement principle.

Discussion:

At several points in the workshop, vendors expressed a preference that the Federal reference method for IP be based on design specifications.

Performance specifications were not particularly appealing since the vendors did not possess the in-house expertise to sit down and design a sampler that would perform adequately. Furthermore, they claimed that the limited market for particulate samplers did not justify the expense of using outside researchers to design and/or test the samplers. Hence, they tended to prefer a design specification approach to the Federal reference method for IP. (The vendors also made the astute point that even if the reference method is based on performance specifications, manufacturers ultimately will be following design specifications for sampler designs that have been developed and tested by researchers. Section 3.2.2 is particularly relevant.) However, results of wind-tunnel testing (Section 3.3.2 and 3.3.3) and field testing (Section 3.3.1) of IP samplers developed to date suggest that, although a design specification approach is simple in concept, it may be difficult to make it effective because it is all too easy to pick a sampler design that fails to perform as expected.

3.2 MEASUREMENTS OF AMBIENT IP CONCENTRATIONS

Prior to EPA's interest in 15-μm inhalable particles, little attention had been paid to sampling large particles in ambient air. Most efforts were directed toward sampling fine particles (say less than 3.5 μm), either because they were considered potentially the greatest health hazard or because of effects on atmospheric visibility. However, Dr. Dale Lundgren reviewed his work reported in 1975 concerning the size distribution of large particles in the atmosphere. In 1979, EPA established an Inhalable Particulate Network to gather IP data and to field-test available hardware. Mr. Charles Rodes (EPA/EMSL) reported preliminary results from this effort.

3.2.1 Large Particles in Ambient Air, Dr. Dale Lundgren, University of Florida

Summary:

Dr. Lundgren introduced his remarks as being a repeat of a 6-year-old presentation.* He presented data emphasizing the particle distributions in

*Lundgren, D.A., and H.J. Paulus, "The Mass Distribution of Large Atmospheric Particles," J. Air Pollut. Control Assoc., 25 (12):1227, 1975.

the 10- to 100-μm aerodynamic diameter range. Using data as early as that of Junge, he showed that atmospheric particles have a bimodal mass distribution with a submicron peak and a peak somewhere between 10 and 100 μm. All sources of data, including data taken by Dr. Lundgren, indicate that there is a significant mass in the 10- to 100-μm range. This portion of the distribution is the one toward which Dr. Lundgren directed his remarks. He reviewed his design and construction of a mass sampler used to investigate the mass distribution of particles in the large peak region. This sampler, placed on a school building roof in semirural Minnesota, consisted of a 60-cm diameter duct to the interior of the building from which five samplers collected particles isokinetically. The first four of these samplers had particulate cutpoints at 60, 30, 15, and 8 μm; the fifth sampler was a total particulate sampler. The flow rate through the main sampling tube was 900 ft^3/min. Sampling was done for 24 hours.

Dr. Lundgren identified much of what he collected as pollen particles; his catch was largely organic. One specific run that he highlighted in the presentation was taken with the wind blowing from a freshly plowed field. For this run, the average concentration was on the order of 60 $\mu g/m^3$ with a mass median diameter (MMD) between 10 and 20 μm. One run could be fitted by summing two distributions: one distribution with a submicron mass median diameter of 0.5 μm, $\sigma_g = 4$ and a second distribution with a mass median diameter of 15 μm and a $\sigma_g = 2$.

In addition to the special sampling filters, a standard high-volume sampler was always run simultaneously; it was shown that the hi-vol sampler collected 97 percent of the total mass collected by the total particulate sampler. These conclusions were consistent with other statements describing the hi-vol cutpoint as being on the order of 60 μm. This clearly shows that picking a 15-μm cut, by the definition of an IP standard, changes the sampling cut from the present TSP standard.

Dr. Lundgren also reported that the density of collected particles seems to be close to 1 g/cm^3, leading to his conclusion that the bulk of the mass was organic. This conclusion was true except for the one run with wind from the plowed field for which the density approached 2 g/cm^3, characteristic of silica particles.

Discussion:

In the question and answer period, the sampling through the 2-ft duct was stated to be without a screen or cover but inside a 5-ft shroud. It included only one 90° bend. Most air flow into the duct came from the top of the shroud. This conclusion was based on smoke tests.

A question asking for a comparison of the Minneapolis data with desert dust data was answered by saying that the desert data are much richer in larger particles (1,000-μm sand can exist in such desert data). No particles greater than 500 μm were seen in Minneapolis, even in the plowed field case.

3.2.2 IP Network Sampling, Mr. Charles Rodes, EPA/EMSL

Summary:

During fiscal year 1979, EMSL attempted to obtain values of both IP and TSP in support of (1) a revised standard and (2) control technology strategy for that standard. Mr. Stevens of ESRL recommended the dichotomous sampler with a cutpoint adjusted to achieve 15 μm as the IP sampler of choice for the network. Recent findings, however, showed that the cutpoint of the conventional bell-shaped inlet of the dichotomous sampler is very windspeed-dependent.

A possible alternative is a modified inlet for the standard high volume sampler. A size selective inlet (SSI), designed for a 15-μm cutpoint, has been demonstrated on a high volume sampler by Dr. McFarland. An alternative then is to retrofit existing high volume samplers with the SSI.

Mr. Rodes then reported on a mathematical analysis* of four types of samplers to determine the effect of inlet cutoff shifts and sharpness on mass measurements assuming typical ambient aerosol size distributions:

1. The conventional high-volume sampler.
2. The size-selective inlet high-volume sampler.

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*This analysis was based on inlet performance measurements made by McFarland. More recent work by Wedding and John (Section 3.3.2), however, indicates a greater wind sensitivity of the dichotomous sampler head, particularly at low wind speeds.

3. The Beckman dichotomous sampler.

4. A new Ben Liu inlet design for the dichotomous sampler (Section 3.3.1).

This analysis was carried out using Dr. Lundgren's 1973 data as typical of the particulate burden to be experienced. Estimates of expected mass collected were made for windspeeds of 2, 8, and 24 km/h for each of these instruments. An immediate conclusion of this analysis was that expected mass collected was not very sensitive to differences in sampler inlet particle separation curves. For example, the calculations showed that a change from a 15-μm cutoff to a 13-μm cutoff resulted in about a 10-percent error, not as bad as might have been expected. The windspeed dependence of some inlets was shown to have more effect on expected mass collected and Mr. Rodes felt the problem should be eliminated.

Field (pilot) testing of the IP samplers was carried out in Durham, NC, and in Los Angeles, CA. A number of samplers were operated side by side: Three from each of three brands of dichotomous samplers, three SSI high-volume samplers, and three conventional high-volume samplers. The 10-m altitude windspeed from local airports was used as the reference air speed. The measured windspeed at Durham was low (less than 2 mph) throughout the study.

In field testing in Durham, the dichotomous samplers proved to be tough to operate and a training effect was clearly evident in the data. The coarse fractions (the 2.5-to 15.0-μm size range) varied much more than the fine fractions (<2.5 μm). The coefficient of variation for the coarse fraction in the Sierra dichotomous sampler was as high as 30 percent, but for the fine fractions, the coefficients of variation were 15 percent for the Sierra and 11 percent for the Beckman. Generally, there is more mass in the fine fraction, so overall the total mass measurement has better reproducibility (~7 to 8 percent)--primarily because the fine fraction dominates. By contrast, the coefficient of variation for the size-selective hi-vol was about 5 percent, which Mr. Rodes considered quite good. The reproducibility of the standard hi-vol was 2 percent.

The data from the Los Angeles study indicated an additional problem: a bias between the dichotomous samplers. Special care in handling and weighing the filters did not eliminate this problem.

Mr. Rodes also discussed flow rate stability problems with dichotomous samplers. He mentioned that a loading phenomenon seemed related to relative humidity. For example, at 85 $\mu g/m^3$, operation is okay at low humidity; but when relative humidity is high, the same loading reduces the flow rate significantly. Loading also depended on the type of filter used. Teflon® filters with nominally 2-μm pore size showed much less tendency to clog than those with 1-μm pore size.

Discussion:

Comparisons of nitrate and sulfate percentages of total mass in IP field test show that artifact formation is probably not a problem.*

Some reasonable fraction of the hi-vol mass appeared on the dichotomous samplers in more or less agreement with the mass collected by the size-selective hi-vol samplers. The IP/TSP ratio was about 0.7 in both Durham and Los Angeles with surprisingly little variation.

Conclusions:

Mr. Rodes stressed two points: (1) the technology for making reproducible IP measurements might not be available yet (although development efforts were underway), and (2) the assessment of IP Network data depended on what level of reproducibility one should expect with ambient particulate measurements. When pressed, Mr. Rodes volunteered 15 percent as an acceptable and achievable level of reproducibility. Not everyone at the workshop agreed with this level, but the limited data precluded more definitive conclusions (see Part II, Section 2.2.1).

3.3 EVALUATION OF INSTRUMENTS FOR MEASURING IP

A number of workshop participants reported results of laboratory evaluation of existing IP samplers. Existing IP samplers use an inertially

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*It was later learned that Gelman microquartz, not glass fiber filters, were used in these field tests and in the IP Network in 1979, which may account for the low artifact percentages. There were an insufficient number of collocated samplers to statistically verify this contention, however.

separating inlet to remove particles primarily greater than 15 μm from the sampled air and then either collect the catch on a single filter or further classify the particles into two fractions collected on separate filters (dichotomous samplers). Much of the evaluation effort focused on establishing the inlet particle separation characteristics. The general approach taken was to expose the sampler to a controlled test atmosphere containing a low concentration of fluorescent-tagged liquid particles. Drs. Liu, Wedding, and John presented evaluations of ambient IP sampler particle separation characteristics. Dr. Harris and Mr. Cushing discussed source assessment of IP and the measurement instrumentation used. Dr. Stevens addressed a different aspect of IP measurement--interferences arising from artifact formation on filter media.

3.3.1 Sampler Effectiveness, Dr. Benjamin Liu, University of Minnesota

Summary:

Liu outlined his presentation as follows:

1. A review of inlet design.
2. A theory listing the important factors for high efficiency sampling.
3. The presentation of a new design and hardware to eliminate non-IP particles from the sample.

An inlet should meet four requirements:

1. Eliminate snow, rain, insects, and debris.
2. Transmit particles to the filter.
3. Eliminate the noninhalable particles.
4. Remain unaffected by windspeed and direction.

Traditionally, inlets have done reasonably well on the first requirement but not so well on requirements 2 through 4. For example, in isokinetic sampling, it is easy to match wind direction, by a wind vane or something similar, but difficult to match windspeed. Dr. Liu then reviewed the existing inlet designs for ambient sampling and made some comments on their performance before presenting his theoretical analysis of the inlet problem.

His emphasis in this review was on the imprecision of design (the Rockwell sampler device, for example, was designed for a 20-μm cutpoint but actually had a 25-μm characteristic) and on windspeed dependence (the Beckman device exhibited a 15.5-μm cutpoint at 2 km/h but shifted to a 10.5-μm cutpoint at 24 km/h windspeed). Sampling efficiency itself is size-dependent. In general, factors such as particle losses on both internal and external surfaces and sensitivity to flow disturbances are not adequately handled as yet.

The starting point for Dr. Liu's design was a 1978 paper by Zebel* who solved the potential flow equations under the boundary conditions of a hole in an infinite wall. Dr. Liu's design concept began with a flanged circular tube. It should show high aspiration efficiency for 15-μm particles as predicted by Zebel's equation. With the addition of an impaction cup to eliminate the coarse particles and a top cover to keep out the rain, the elements of his new candidate IP sampler emerge.

In measuring performance of the sampler, Dr. Liu chose to define three separate efficiencies:

1. The aspiration efficiency = M_1/M_o.
2. The transmission efficiency = M_2/M_1.
3. The overall efficiency = M_2/M_o.

where

M_1 = the mass exiting the inlet nozzle plane.

M_o = the mass in the ambient atmosphere.

M_2 = the mass exiting the plane of the inlet.

Initial testing of his sampler showed a very dramatic drop in aspiration efficiency with windspeed, while the impactor transmission efficiency was independent of windspeed and had the following properties: D_{50} = 13.3 μm, σ_g = 1.18. Zebel's theory predicts only a small shift in overall

*In Recent Developments in Aerosol Science, Shaw, ed., New York: John Wiley & Sons, 1978.

efficiency as windspeed increases to 24 km/h. By increasing the diameter of the hole in the infinite plane, Dr. Liu achieved performance close to that predicted.

The shape of the impaction cup shifted the cutpoint to 13.3 μm rather than the 15 μm specified in the design. The cup is designed to make multiple collisions more probable for bouncing particles; particle bounce and penetration of large particles do not seem to be a problem with this design.

Wall losses are typically 3 percent or less and never exceed 8 percent. Dr. Liu cautioned against housing interferences. The placement of the sampler inlet should be sufficiently far from its own housing to minimize such interferences; alternatively, the housing should include a flat plate to simulate an infinite plane adjacent to the inlet.

Discussion:

The question of freezing rain and water entry into the inlet was answered by citing the ease with which a drain could be put into the impaction cup. Dr. Liu further claimed that the inlet would not be affected much by sedimentation.

In response to an earlier comment regarding design of inlets, a Beckman representative raised the question of impaction losses on the lip of the Beckman dichotomous inlet. Dr. Liu replied that test data for the Beckman sampler did show a shift of cutpoint with windspeed, but whether this effect resulted from impaction losses on the lip had not been determined.

Conclusion:

Liu's inlet was designed from first principles of aerosol mechanics. Subsequent testing of this original design, however, showed that the inlet was susceptible to particle bounce problems (see Part II, Section 2.1.2). This result underscores the point of view of the vendor community that designing a sampler to conform to certain performance specifications for sampling large particles is not a straightforward task. At the same time, it also points up the danger in designating a particular sampler design as the reference method if that sampler has not been very well characterized.

3.3.2 Wind Tunnel Testing of Dichotomous Samplers, Dr. James Wedding, Colorado State University

Summary:

Dr. Wedding's presentation was the first of two emphasizing the variation of sampling efficiency of existing dichotomous samplers at low windspeeds. The fall-off in efficiency at the low windspeeds, attributable to sedimentation losses, had not previously received attention. His presentation concentrated on what he interpreted to be boundary layer separation effects at the lower end of the windspeed spectrum. He agreed that the top end of the windspeed ranges were solved.

He had tested both Sierra-type and Beckman-type dichotomous samplers in the Colorado State University test facility. Using oleic acid-uranine aerosol, he found cutoffs highly dependent upon windspeed.

Dr. Wedding also described a new experimental inlet that used a cyclone to achieve the IP cut. The prototype had minimal particle bounce problems and achieved a sharp cut at 15 μm (σ_g = 1.3, 1.4). Efficiency for 15-μm particles in the 2- to 7-km/h ranges was flat; above 20 km/h, impaction losses predominated. Further modifications to this inlet reduced these losses to acceptable levels, however.

After rough calculations by a number of participants, it was generally decided that settling losses were not important in the Liu inlet and the Wedding experimental inlet for the low windspeed conditions.

Conclusions:

The bell-shaped inlets used on both the Sierra and Beckman dichotomous samplers apparently failed to achieve the desired cutpoint of 15 μm based on tests of liquid particles in a wind tunnel at low windspeeds. Later testing by Walter John revealed that the bell-shaped inlet could be made to cut at 15 μm simply by increasing the inlet gap (See Part 2, Section 2.1.2).

3.3.3 Further Evaluation of a Dichotomous Sampler, Dr. Walter John, California Department of Health Services

Summary:

Dr. Walter John carried out static inlet tests by placing the unit

under test (a Sierra dichotomous sampler) in a big plastic bag. Oleic acid-uranine particles were fed into the top of this bag and permitted to settle so that the zero windspeed condition was simulated within the bag. In this sampling arrangement, excess air simply overflowed.

Dr. John found that whereas in the wind tunnel the cutpoint of an existing dichotomous sampler increased to 22 μm as the windspeed decreased, under static conditions the cutpoint decreased to 11 μm. The sampling efficiency curve was very steep in the 0- to 2-km/h range.

At one point, Dr. John replaced the hat of the dichotomous sampler with a lucite top in order to study the flow within. By using smoke patterns, he was able to show that the inlet gas simply flows up the ramp and that only a fraction of the inlet gas makes it to the sampling port. He performed a calculation from first principles using gravity and centrifugal forces to predict efficiency versus windspeed and obtained a curve with a sharp maximum between 0 and 2 km/h. He concluded that although the operation of a sampler is known both theoretically and experimentally, it is clear that the operation is unsatisfactory at low windspeeds and he saw a need for a better inlet design and testing under static conditions.

Discussion:

The possibility of correcting dichotomous sampler data for variation in cutpoint with windspeed was raised. It was pointed out that the cutpoint variation was very sensitive to windspeed; consequently, it would be necessary to measure the windspeed very accurately, particularly low windspeeds. But this measurement was not considered easy either, especially with common, inertial wind-measuring devices such as cup anemometers. An alternative position and one that seemed to win the most favor was to ignore the problem since low windspeed conditions constituted only a small fraction of the total data.

Conclusions:

These results and subsequent discussion raise an important issue: even if laboratory evaluation of a sampler indicates unacceptable performance, what is the significance of this behavior when sampling ambient air? Here, for example, the observed jump in sampler cutpoint at low windspeeds

may not significantly affect the IP catch when it is realized that these conditions may arise infrequently. Furthermore, the concentration of large particles is likely to be low during low windspeed conditions. The issue remained unresolved, but it was clear that a sampler should undergo some testing in ambient air.

3.3.4 Artifact Formation, Mr. Robert Stevens, EPA/ESRL

Summary:

Positive artifacts consist of sulfates and nitrates formed by gas-to-particle conversion, which occurs on certain filter media. Mr. Stevens reviewed the literature on the subject, including negative artifact formation (loss of material) reported just this year and presumably caused by nitrate volatilization and/or chemical reactions. Artifact formation represents an interference in measurement of aerosol mass.

He then listed the composition and pH of typical filter media and presented data to show that artifact formation is most pronounced with glass fiber filters. He concluded that filters can distort the sulfate measurements but probably do not significantly affect TSP. His rough number range was as follows:

1. SO_2 reacts with glass fiber filters to form 0.1 to 5 $\mu g/m^3$ artifact;

2. HNO_3 and NO_2 react with glass fiber filters to form as much as 50 $\mu g/m^3$ of artifact in photochemically active areas, but more typically 0.1 to 5 $\mu g/m^3$;

3. Teflon®, quartz, and Nucleopore filters do not produce positive sulfate or nitrate artifacts.

Mr. Stevens also identified a third type of artifact that he attributed to acid organics. Again he postulated a reaction between organic acids and substances on the glass fibers to form a new artifact. An overall conclusion emerging from his discussion was that it may be wise to change the filter material if the IP Federal reference method allows a modification of the existing high-volume samplers to serve as a reference method.

Points to consider include:

1. In Mr. Stevens' opinion, artifacts would be unimportant even with glass filters everywhere except in specific hot spots, especially in California.

2. Tentatively, he recommended forgetting artifacts unless sulfates or nitrates exceeded 10 percent of TSP.

3. He cautioned, however, that a switch in cutpoint to 15 μm would reduce the mass catch for IP to 30 to 50 percent of the TSP catch. Artifact contributions that might be negligible at the original TSP standard then could become more significant.

Discussion:

Dr. Bruce Harris recommended Reeve Angel glass fiber filters, which he had found to work well for stack samplers, and offered the opinion that they may also be appropriate for ambient air sampling. ("Work well" presumably means remained stable under time and temperature of stack sampling and showed negligible artifact formation due to gaseous interactions.) It was Dr. Harris' contention that the driving force for gas-to-particle reactions would be greater at higher temperatures. The suitability of these filters would depend on the amount of artifact relative to the loading encountered in ambient sampling, however.

There was some discussion concerning Teflon® filters. Ben Liu requested that the filter specifications for Teflon® filters include a pore size specification and not just a nominal size determined by flow characteristics. James McDaniel pointed out that the pore size could be controlled by measuring the pressure drop across the filter. Walter John added that the efficiency of the filter is always correlated with the pressure drop. Rodes mentioned the high cost of Teflon® filter, $1.50 to $2 each. In general, a number of voices acknowledge that company-to-company and filter-to-filter variations exist, so that it may be necessary to better control the allowed variations.

Conclusion:

The issue of which filter material(s) could be used in the reference method for IP was not resolved. Allowing flexibility in sampler design suggested that flexibility also be allowed in choice of filter medium. Glass fiber appeared a poor choice, but Teflon® had a drawback of high

cost. The general feeling was that data gathered by the IP network would help in the assessment of artifact problems and choice of filters.

3.3.5 Source Level IP, Dr. Bruce Harris, EPA/IERL; Mr. Kenneth Cushing, Southern Research Institute

Cyclone IX operating at 14 L/min and 150° C falls neatly within the proposed IP performance envelope. Mr. Cushing had data showing that the flow rate needed to get a 15-μm cut varied with temperature. Efficiency decreased with temperature and also varied with viscosity. He proceeded to show some efficiency calculations for a horizontal elutriator with either a rectangular or circular channel and included a theoretical calculation for the IP performance specification.

The last afternoon session began with a presentation from Dr. Harris on artifact formation or filter interferences in collecting stack samples. Dr. Harris indicated that the Reeve Angel 934, a John Mansfield material, showed no mass increase with conditioning time (up to 9 hours) and it was unique among the filters he had tested in this respect. In response to a question, he stated that this was an untreated filter, one which had not undergone any acid wash or other preparation. Some filters had been found to be more stable following a wash treatment of 50 percent sulfuric acid, isopropyl alcohol, and water.

The impactor used in stack sampling has an 8- to 10-μm cutoff and Dr. Harris repeated the observation that if the health people had accepted the existing 8- to 10-μm cutoff instead of insisting upon 15 μm, a lot of IP emission data would already exist. As it stands now, the cutoff is of dubious value, although attempts will be (and have been) made to extrapolate IP data from existing impactor data. Actually, it is an interpolation since one can use the impactor data plus the first-stage catch as an end-point and somehow connect the existing 10 μm with the first-stage catch and deduce what the 15-μm cutoff mass would be.

Dr. Harris also showed sketches of a cyclone inlet to modify the cascade impactor into an IP cyclone sampler. A 15-μm cutoff can be obtained; indeed one can have a two-stage cyclone, the second cyclone having a 2.5-μm cutoff, should that prove to be of interest later.

3.4 DEVELOPING PERFORMANCE TESTS FOR CANDIDATE IP SAMPLERS

Assuming a performance specification approach to the IP Federal reference method is adopted, a number of questions immediately arise. Which performance parameters need to be tested? How shall the tests be performed? And what should the acceptance levels be to ensure that different sampler designs that pass the performance tests will also give comparable ambient measurements? Mr. Kashdan considered technical aspects of aerosol generation in regard to performance testing. Dr. Ranade addressed himself to the protocol and acceptance levels for tests of the sampler inlet. Dr. McFarland discussed requirements for a wind tunnel to evaluate sampler particle separation characteristics. Dr. Loo reviewed his work in performing static tests of samplers, in particular, laboratory and field evaluation of the dichotomous sampler.

3.4.1 Aerosol Generation, Mr. Ed Kashdan, Research Triangle Institute

Mr. Kashdan reviewed aspects of aerosol generation involved in evaluating candidate IP samplers. In particular, he directed attention toward the testing of sampler (inlet) effectiveness--the particle collection efficiency of the sampler relative to an isokinetic probe. In the past, this test had been performed in a wind tunnel using monodisperse liquid particles that were tagged with a fluorescent dye and generated with a vibrating orifice aerosol generator. Incorporating this into the testing requirements for IP samplers, however, requires standardizing and detailing the procedures.

Mr. Kashdan outlined the considerations involved in standardizing the sampler effectiveness test and suggested a tentative approach. The first consideration was the specifications for the aerosol generator--simply require a vibrating orifice generator or specify a standard of performance. A monodispersity specification of $\sigma_g = 1.1$ for the aerosol generator was suggested. Second, he proposed a particle size range of 5 to 30 μm. The justification for this range was that fine particles below 5 μm would follow the streamlines and were not likely to be collected by an inertially separating inlet cutting sharply at 15 μm; particles greater than 30 μm would not only be difficult to maintain suspended in a wind tunnel but might not represent a realistic situation. Third, he proposed testing equally with solid and liquid particles--solid particles were necessary to

test for particle bouncing. The specter of using solid particles raised unresolved questions of which test aerosol to use and how to conveniently analyze it, since large, solid aerosols could not be tagged easily with a fluorescent tracer. Finally, checking the size of liquid particles with a microscope was proposed as a quality assurance measure.

Discussion:

All the aspects of aerosol generation considered by Mr. Kashdan received some discussion. In regard to the aerosol generator, Dr. Lundgren indicated a preference for a spinning disk aerosol generator, which produced greater quantities of aerosol than the vibrating orifice generator. Dr. Liu pointed out that the vibrating orifice generator is closest to a primary standard for particle size and therefore would not require calibration; further, he argued against attempting to calibrate the vibrating orifice generator against a microscope because of the need to use a flattening factor to correct the particle size measured under the microscope. Lundgren took exception to this argument by emphasizing that the accuracy of the vibrating orifice settings was limited by the need to dry the particles adequately--a problem he experienced even at Dr. Liu's course in Minnesota.

Mr. Stevens and Dr. Wedding argued for including particles below 5 μm in the test procedures, especially if a dichotomous sampler is used. Dr. Wedding also argued for including large particles since in his experience some samplers showed an increase in efficiency as the aerodynamic size exceeded 20 μm. The benefit of additional testing must be weighed against the cost--Dr. Liu pointed to a large factorial matrix (of wind speeds and particle types and sizes), which would be quite costly to conduct.

The question of solid vs. liquid particles also received discussion. Dr. Dzubay argued for a limited factorial design that, if carefully planned, could maximize the information without testing at every combination of windspeed and particle size and type. A related issue was which aerosol to use in a solid particle test--some participants thought that a well-characterized, polydisperse dust would be ideal especially if it could abbreviate the testing protocol by testing several sizes simultaneously. A variety of materials were suggested for the solid test aerosol including Arizona road dust, flyash, glass beads, polystyrene beads, and pollens.

Dr. Walter John cautioned that the size distribution was strongly dependent upon dispersion technique and that, in general, solid particle size distributions are not reproducible. Dr. Lundgren characterized the problem of inlet effectiveness as one of primarily restricting the large particles and suggested a single test with one source of solid particles. Analysis of collected solid particles was also seen as a problem, as counting under a microscope is tedious and weighing may require long generation times or high concentrations. Lastly, Mr. Stevens and Dr. John argued for using a real-time particle monitor, such as an optical particle counter, to monitor the performance of the aerosol generator.

Conclusion:

Obviously, the sampler effectiveness test could be made quite extensive and costly. Practical concerns, however, dictate a limited experimental design. The design that seemed to appeal to most participants was to test with liquid particles in a 5- to 25-μm range and to test for particle bounce with one large size of a solid aerosol. It was argued that since the likelihood of a particle bounce problem was dependent on the momentum of the particle, a sampler that did not show particle bounce problems at 30 μm probably would not have problems with smaller particles. Testing with 1-μm liquid particles did not seem necessary as long as only the inlet was being evaluated. The choice of solid test aerosol material remained unresolved.

3.4.2 Performance Specifications, Dr. Arun Ranade, Research Triangle Institute

Dr. Ranade began by restating the preference for an IP Federal reference method based on performance specifications. He directed attention toward the windspeeds at which the inlet effectiveness test should be performed and the acceptance levels for this test.

The average annual U.S. windspeed is 15 mph (24 km/h), which Dr. Ranade claimed could serve as a top speed requirement for the wind tunnel. As for a lower limit on windspeed, his recommendation was to use some low speed to simulate quiescent conditions in the atmosphere, but an absolutely static test at zero windspeed, he thought, would be difficult to implement and of questionable value.

The next topic was the specification of an acceptance "envelope" for the inlet effectiveness test. The envelope, a region on a graph of effectiveness versus particle size, was defined by lognormal distributions with parameters MMD = 15 ± 2 μm and σ_g = 1.0 - 1.7 (see Figure 1, Appendix C). The original concept of an envelope arose out of a contractors meeting* in which a σ_g of 1.5 ± 0.2 was used and presumed to represent the performance of existing inlets. But the possibility of an ideally sharp-cutting inlet required the range to be expanded to include a σ_g down to 1.0.

Dr. Ranade also mentioned a performance test of flow rate stability. He envisioned placing the sampler in ambient air and maintaining the indicated flow rate within tolerance limits over 24 hours. Values of the tolerance limits were not specified.

Discussion:

The first topic to provoke discussion was specification of the acceptance envelope for sampler effectiveness. Two aspects were considered: how best to specify the envelope and how samplers that have acceptable sampler effectiveness curves compare on indicated IP mass concentration. Dr. Walter John emphasized that the effectiveness curves for dichotomous samplers were not lognormal, so it may be unrealistic to even argue the specifications in these terms. Furthermore, if a lognormal specification is used, he argued for a size range dependence for the specified σ_{gs} (sampler σ_g) since it is comparatively easy to get a small σ_{gs} at the large end but difficult at the low end of the particle size spectrum. Dr. Lundgren agreed: have a tight σ_{gs} for particles greater than 15 μm (perhaps a 1-percent penetration at 30 μm) but allow a σ_{gs} up to 1.7 for small particles.

Both Dr. Harris and Dr. Liu attempted to evaluate sampler effectiveness in terms of expected IP mass collected. (The approach was analogous to that used by Mr. Rodes, Section 3.2.2, except the input distributions were different.) Dr. Harris, using various input lognormal distributions, concluded that σ_{gs}, varied even from 1.0 to 1.7, did not make a large

*Southern Research Institute, "Sampling Techniques for Inhalable Particulate Matter," Report SoRI-EAS-79-031 under EPA Contract No. 68-02-2612, Birmingham, AL, January 1979.

difference in the expected IP catch. Dr. Liu, using similar calculations, chose a worst-case input distribution (MMD on the order of 25 μm) and found a 30-percent difference in IP mass between a sampler having an inlet σ_{gs} of 1.7 and one having a σ_{gs} of 1.0. Input distributions with smaller MMD's showed less difference in IP mass. However, Dr. Liu pointed out that a smaller σ_{gs} also produces an inlet of reduced windspeed dependence.

Dr. Dzubay brought up the question of flow rate stability. If the sampler flow rate does not remain stable over the 24-hour sampling period, not only will the sampled volume be incorrect, but the cutpoint will vary. He cited the classic example of the sampler that worked beautifully as long as the ambient dust loading was 75 $\mu g/m^3$, but clogged immediately and failed to operate when exposed to episodes at e.g., 300 $\mu g/m^3$. He questioned whether a flow rate stability test would adequately cover these two cases. Mr. Stevens recounted his experiences with Teflon® filter clogging and pointed out that aerosol composition, not just concentration, was important. He stressed that the percentage of fine particles strongly influenced filter clogging. A test of flow rate stability, even at two ambient concentrations, might therefore not be conclusive.

Various suggestions emerged for solving the problem of flow rate stability. Some participants wanted a performance specification in terms of either flow rate or pressure drop. Dr. McFarland proposed a laboratory performance test wherein filter loading would be simulated with a valve on the inlet. Other participants brought up design measures including a cutoff switch, a filter changing mechanism, and a bigger pump. With a cutoff switch, the sampler would be capable of shutting down when the pressure drop exceeded a certain predetermined value. The sampler would also be able to record the time at which the shutdown took place so as to yield a useful measurement of concentration over that period of time during which it did operate. This design measure had a recognized drawback in that it failed to provide a 24-hour measurement and failed perhaps during an episode when it would be most desirable to have a measure of population exposure. A filter-changing mechanism obviated this problem, but only with an increase in the cost of the sampler. Specifying a large pump (larger capacity than one that had been shown to have problems maintaining sampler flow rate) was not considered adequate by all attendees; some felt that the

additional pressure drop would affect flow measuring devices while others claimed that no air could be drawn through a totally clogged filter regardless of pump size.

Dr. Liu had reservations about a performance test of sampler reproducibility (a test of the capability of duplicate samplers to obtain comparable results when operated side-by-side in ambient air). He raised the question of the importance of the test site. He said Los Angeles, for example, would make it particularly easy to qualify. Dr. Liu also added that the position of the samplers with respect to each other and obstructions is a crucial factor in the sampler performance. He recommended that the guidelines specify geographical locations as well as dimensions and clearances from obstructions.

Conclusions:

Dr. Ranade displayed the performance envelope bounded by lognormal distributions of MMD = 15 ± 2 μm and σ_g = 1.0 - 1.7 and solicited opinions in an open discussion. The general consensus seemed to be that a 10-percent error band at the lower size range should be added for experimental error (see Figure 1, Appendix C). At the large particle end, there were differences of opinion--some people felt that a 10-percent error band was acceptable, while others felt that 10 percent penetration was out of the question. It was clearly understood that large particles contribute heavily to the aerosol mass collected. Even though no decision was reached on the acceptance level for large particle penetration through the inlet, testing with large particles, particularly "bouncy" solid ones, should be required.

There is a strong interplay among laboratory performance tests, performance tests in ambient air, and ambient IP measurements. Laboratory performance tests (e.g., inlet effectiveness test) can be conducted under controlled conditions and can provide a characterization of sampler performance. As evidenced by the work of Rodes, Liu, and Harris, it was generally acknowledged that the laboratory characterization should be expressed in terms of expected performance in ambient air (e.g., expected IP mass collected). To further characterize expected performance, the workshop group considered tests of flow rate stability and sampler reproducibility under ambient conditions. But because conditions would be uncontrolled and

practical concerns would permit only limited testing, such tests might not always be representative of sampler performance. Ambient IP measurements from the IP Network represent ambient performance testing on a large scale; as discussed by Rodes (Section 3.2.2), however, the data were not particularly conclusive because of the many uncontrolled variables. The general consensus of the workshop group was that after sufficient data had been gathered by the IP Network, acceptance levels for the ambient performance tests (flow rate stability and sampler reproducibility) could be confidently set.

3.4.3 Wind Tunnel Tests, Dr. Andrew McFarland, Texas A&M

Dr. McFarland described the testing of various samplers he performed in his wind tunnel facility. He used a vibrating orifice to generate liquid particles at the wind tunnel entry and collected test data at windspeeds from 2 to 24 km/h. Dr. McFarland checked the size of the generated particles by microscope, but he also recommended using an optical particle counter as a real-time monitor of the generator.

Among the samplers he had tested was the Beckman dichotomous sampler. As was the case for the Sierra dichotomous sampler (Section 3.3.2), the inlet of the Beckman showed a dependence on windspeed: the cutpoint dropped from 15 μm at 2 km/h to 10 μm at 24 km/h.

Comments made by Dr. McFarland concerning using a wind tunnel for sampler performance testing were:

1. There is no such thing as a universal wind tunnel test facility: the size of the wind tunnel necessary to test a particular sampler depends on the size of the sampler. Placing a dichotomous sampler inlet in a very large wind tunnel (suitable for testing a size-selective hi-vol, for example) only necessitates longer aerosol generation times but did not improve the evaluation. For one particular sampler inlet, he had found that a large change in wind tunnel cross section, from 25 ft^2 to 64 ft^2, had only a slight effect on the determination of the cutpoint.

2. No value for the concentration of aerosol to be used in the wind tunnel needs to be specified. The concentration simply has to be high enough to obtain a filter catch that can be analyzed with good sensitivity in a reasonable amount of time.

3. Turbulence intensity levels within the wind tunnel need not be specified--just a mass uniformity specification. He recommended a 10-percent variation only in the center zone of the wind tunnel, which he defined as the sampling zone.

Discussion:

Dr. Wedding put forth two criteria that could be used to specify requirements for a wind tunnel for testing IP samplers. First, to specify the size of the wind tunnel, he cited a guideline that required the test body (sampler inlet) cross section to be no greater than 5 percent of the wind tunnel cross section. Second, as for minimum wind tunnel speed, he said the lowest practical speed is 0.5 km/h. Further, for each particle size, he recommended a minimum windspeed in the wind tunnel of 10 times the settling velocity of the particle.

Conclusions:

The emerging message was that, for purposes of testing IP samplers, a wind tunnel need only deliver a uniform particle cloud and be of sufficient cross section that the sampler inlet block a small fraction of the tunnel cross section. The windspeed range should extend to at least 24 km/h (15 mph), the U.S. annual average windspeed, and perhaps down to 0.5 km/h. It might be noted that allowing flexibility in the design of the wind tunnel would be exceptional among standards that require a wind tunnel; e.g., in the ASHRAE test for filter efficiency (ASHRAE 52-76), details of the wind tunnel design are provided.

3.4.4 Static Tests, Dr. Billy Loo, Lawrence-Berkeley Laboratory

Dr. Loo described static and field tests of the Beckman dichotomous sampler. He added that it might be difficult to maintain uniform aerosol concentration in a wind tunnel long enough to complete a satisfactory test run. His setup was reminiscent of the work done by Dr. John (Section 3.3.3), except that he defined a static test as one in which the net flow into the chamber does not exceed what the sampler pulls so that there was no excess air. He used a vibrating orifice generator, which he regarded as the single most reliable particle generator available. The calculated value of particle size was his recommendation for the primary size measurement. He

stated that it could be calculated to within 0.1 μm. Some other backup sizing technique should be secondary.

Dr. Loo's data showed that the alignment of the injecting nozzle and the collecting nozzle in the dichotomous sampler is critical. He claimed that the alignment should be within 1 to 2 mils; a 1-mil error implied a 1-percent loss. He offered criticism of the Beckman design, because, in taking his laboratory model and converting it into a production item, Beckman had seen fit to replace a single rigid member with three separate pieces joined by O-rings. Dr. Loo claimed that Beckman's errors in alignment could be as large as 0.008 inch with 0.004 inch being typical. Therefore, one could see asymmetrical particle deposition on the collection probe of the Beckman dichotomous sampler. Dr. Loo's conclusion is that using two O-rings to align the collector and nozzle is simply poor engineering. (Beckman engineers in attendance had not heard of this criticism prior to this presentation; they expressed not only surprise but appreciation for this bit of information.)

Dr. Loo also described field tests of the dichotomous sampler in the Charleston intercomparison study. A large selection of samplers including a beta absorption sampler, a hi-vol sampler, and both automated and manual dichotomous samplers were placed on a rooftop in Charleston, West Virginia. A side-by-side comparison of 8 days' operation with a measurement every 12 hours was carried out for a total of 16 data points. Any sampler for which less than 8 data points existed was discarded. Sulfate and lead components were also measured.

The results of the sampler comparisons generally showed that the dichotomous samplers had the best precision (as a group, as good or better than any others).

Discussion:

The question of using static tests to evaluate sampler effectiveness received discussion. Dr. John (Section 3.3.3) had concluded that the cutpoint of the Sierra dichotomous sampler reached a sharp maximum as the windspeed decreased from 2 km/h--a characteristic that apparently had not been observed in previous wind tunnel tests. Yet the prospect of including static tests along with wind tunnel tests as part of the reference method

acceptability tests was met with reservations. Dr. Ranade thought that static tests would be difficult to carry out. Dr. Wedding questioned the technical merits of a static test and brought up the possibility of enrichment (sampler collects more than the isokinetic probe) because of settling. Still others questioned the usefulness of a static test as a predictor of sampler performance in the ambient air.

Conclusions:

Although a static test might be useful in the development of sampler inlets, the general concensus of the Workshop group was that static tests probably do not need to be included in tests of candidate samplers. A wind tunnel test at a low windspeed of perhaps 0.5 km/h was seen as not only a reasonable alternative, but perhaps a more realistic test.

PART II

SUMMARY OF THE NOVEMBER 1980 WORKSHOP ON THE FEDERAL REEFERENCE METHOD FOR DETERMINATION OF INHALABLE PARTICLES

SECTION 1

INTRODUCTION

From November 18 to 20, 1980, a second workshop concerning the development of the Federal reference method (FRM) for determining inhalable particle (IP) concentrations in the atmosphere was held in Fort Collins, Colorado, at Colorado State University. As discussed at the 1979 workshop, if the reference method is to be based on performance specifications, each qualified sampler design will have to pass certain minimum wind tunnel and ambient tests. The purpose of the 1980 workshop was to prepare a draft of standardized test procedures that represented the best available thinking.

The workshop was coordinated by Research Triangle Institute [M. B. (Arun) Ranade and E. R. Kashdan] under sponsorship by EPA's Environmental Monitoring Systems Laboratory (K. Rehme and L. Purdue). It was hosted by Dr. James Wedding of Colorado State University. Because of the narrow scope of the workshop, attendance was limited to persons engaged in the development and/or testing of IP samplers and to persons involved in writing the reference method. A list of attendees is presented in Appendix B.

SECTION 2

SUMMARY OF THE IP WORKSHOP

Attendees made presentations on several aspects of IP sampling, including the status of the IP Standard, inlet development and testing, and artifact formation on filter media. These presentations are summarized in Section 2.1, with emphasis on how these results might be used in the formulation of the Federal reference method for inhalable particles. Following the presentations, the workshop group discussed procedures for performance testing of IP samplers and critiqued a draft of tentative testing procedures (see Appendix C) that had been prepared and distributed to all attendees prior to the workshop. This group discussion is summarized in Section 2.2 according to several aspects involved in sampler testing including performance specifications, aerosol generation, and wind tunnel requirements.

2.1 REVIEW OF DEVELOPMENTS IN AMBIENT IP SAMPLING

2.1.1 Status of the IP Standard

Larry Purdue opened the workshop with a discussion of the status of the development of an Inhalable Particulate Standard. It was obvious that EPA would not meet its planned deadline of December 1980 for promulgation of a new standard. It was uncertain at this time at what cutpoint the standard might be set, but 15.0 μm seemed the most likely. A fine particle standard (0 to 2.5 μm) was a possibility as well. The best course was to proceed with the development of testing procedures for IP samplers that would be applicable regardless of size range.

2.1.2 IP Sampler Design and Testing

Drs. James Wedding, Andrew McFarland, Walter John, Billy Loo, and Benjamin Liu presented results concerning either the development of new inlets or testing of old inlets. Drs. Wedding and John described two innovative bell-shaped inlets. Dr. Wedding's design incorporated a cyclone to obtain a sharp IP cut and eliminate windspeed dependence. The Wedding

inlet had been tested with potassium biphthalate aerosol in a static chamber and had been found to be free of particle bounce for particles up to 20 μm. Dr. John's design used an impaction stage to achieve a sharp IP cut; a porous plate through which oil was drawn by capillary action prevented particle bounce off the impaction surface. Dr. John also pointed out that the original Beckman bell-shaped inlet, which used elutriation to achieve the IP cut, could achieve a sharper cut simply by increasing the inlet gap to 6 cm.

Dr. McFarland presented results of the testing of the OSHA vertical elutriator, which is designed to have a cutpoint of 15 μm; with oleic acid aerosol, he found that the cutpoint was 8 to 10 μm and that the sampler was windspeed dependent. Dr. McFarland also presented data that showed that the 11-1/2 in. × 14 in. and 15 in. × 15 in. hi-vols performed identically in the wind tunnel. Dr. Loo and Dr. Liu described recent design modification to their inlet and presented results. The original (funnel-shaped) design had been modified with a wind deflector inside the impaction cup and a rain guard at the entrance. The cutpoint for liquid particles ranged between 14.3 and 16.5 μm for windspeeds of 1 to 24 km/h. The Loo-Liu inlet had also been tested with sodium chloride aerosol (20 to 30 μm) and glass beads (25 to 35 μm). Particle bounce was a problem with the elastic glass beads, 30 percent of which were reentrained and deposited as wall losses (10 percent were received at the filter).

Dr. Loo presented data from a side-by-side inlet comparison study in Durham, NC, conducted by EPA/ESRL. (See Appendix D for a presentation of data from this study.) Although inconclusive because of a limited experimental design, the results of this study suggested that, in the atmosphere, the Wedding inlet gave comparable results to the conventional Beckman dichotomous sampler inlet even though wind-tunnel tests had shown the latter to be windspeed dependent. This result cast doubt on the capability of wind-tunnel tests to predict the behavior of the samplers in the atmosphere. Mr. Charles Rodes added to this doubt with data from the IP Network. In 60 to 70 percent of the Network sites, the size-selective hi-vol indicated IP concentrations that were consistently 15 to 20 percent higher than the dichotomous sampler concentrations, although this result was not expected on the basis of previous wind tunnel tests. He suggested that positive

sulfate and nitrate artifact with the size-selective hi-vol, and negative nitrate artifact with the dichotomous sampler, however, would account for at least some of the difference.

Most aerosol experts at the workshop felt that inlets previously had not been adequately characterized with respect to particle bounce. Consequently, additional wind tunnel testing with solid particles for particle bounce would resolve the discrepancy between ambient and wind-tunnel performance. Some skepticism remained, however, that unaccounted factors might be contributing to, if not causing, the discrepancy.

2.1.3 Artifact Formation on Filter Media

A determination of the inhalable particle concentration in the atmosphere requires not only a separation between inhalable and noninhalable particles, but also an accurate measurement of the mass of collected inhalable particles, which is most often made by collecting the particles on a filter and weighing them. Artifact formation on the filter surface represents an interference in this mass measurement. Positive artifact formation (gas-to-particle conversion) causes a spurious gain in and consequent overestimation of mass; negative artifact formation (loss of collected particles by evaporation or chemical reaction) causes an underestimation of the mass.

Dr. Bruce Appel presented results of his studies of artifact formation on various filter media. (A more detailed summary is given in Appendix E.) Glass fiber filters are subject to both positive artifact sulfate and positive artifact nitrate errors. Positive artifact sulfate was estimated as up to 12 percent of the IP mass; in areas other than the LA Basin, positive artifact nitrate was estimated as up to 6 percent of the IP mass. Apparently, glass fiber filters are good "total" nitrate collectors, collecting both particulate nitrate and gaseous nitric acid (positive artifact). Because of anomalously high levels of nitric acid in the LA Basin, positive artifact nitrate on glass fiber filters in this region was estimated as up to 18 percent of the IP mass. Teflon® filters do not exhibit sulfate artifact but are subject to negative nitrate artifact interference. This is a particularly difficult problem because a varying and unpredictable quantity

of particulate nitrate may be lost.* Organic artifact may be a more serious problem than either sulfate or nitrate artifact but the data are inadequate to draw conclusions.

2.2 PROCEDURES FOR TESTING CANDIDATE IP SAMPLERS

Following presentations, the workshop group proceeded to a round-table discussion of a number of topics involved in IP sampler testing including the specification of performance characteristics, aerosol generation, wind tunnel requirements, and nomenclature. The following summary is an attempt to present the consensus of the group on these aspects of sampler testing. For some controversial topics, no consensus was reached; in these cases, majority and minority views are given.

2.2.1 Performance Specifications

The tentative sampler testing procedures (Appendix C) require tests of sampling effectiveness, reproducibility, and wall losses. The sampling effectiveness test, performed in a wind tunnel, compares the collection of particles by the sampler to collection by an isokinetic sampler for various sizes of particles at different windspeeds. (See Section 2.2.5 for a discussion of alternative terms used to describe this test.) Acceptance or rejection of the particular sampler on the basis of this test depends on the sampler performance relative to a "performance envelope," an acceptance region of sampling effectiveness versus particle size. It has become the practice to specify this region by lognormal distributions with parameters MMD = 15 ± 2 μm and σ_g = 1.0 - 1.7.† The sampling effectiveness test is designed to evaluate the particle separation characteristics of samplers near the IP cutpoint (15 μm) and, therefore, relatively large particles (>5 μm) are used.

In addition to the test for sampling effectiveness, the workshop group also discussed tests for reproducibility and wall loss. The test for

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*No estimates of the magnitude of the negative nitrate artifact problem were made.

†See also Part I, Section 3.4.2, and Appendix C.

reproducibility involves an ambient comparison between identical samplers and is designed as a quality control check on the precision of the sampler. The wall-loss tests could be incorporated into the sampling effectiveness tests when liquid particles are used. Internal parts of the sampler are either swabbed or washed to recover any aerosol that should have been collected on a filter but was deposited elsewhere.

Performance Envelope. Most attendees seemed to feel that specifying the performance envelope as a composite of two lognormal distributions was acceptable provided that ±10-percent band on the fine particle end was included to account for experimental error and enrichment. (The term "enrichment" refers to the proportionately greater amount of aerosol collected by the sampler relative to the isokinetic sampler. Presumably, it is an anisokinetic sampling effect.) A tentative value of +5 percent at the large particle end was considered acceptable provided that calculations using model aerosol mass distributions (e.g., lognormal or Lundgren's data in Part I, Section 3.2.1) showed that indicated IP concentrations would not be greatly affected by this acceptance level.

Reproducibility.* It was generally agreed that an acceptance level of ±15 percent for the reproducibility between identical samplers operating side-by-side in the field was reasonable. EPA statisticians needed to examine this test to determine the number of samplers and the number of days of testing. Some aerosol researchers present at the workshop felt that a more elaborate test involving an intercomparison among different samplers at various locations would be desirable. However, because of the expense and complexity of that kind of testing, only the more limited test between identical samplers was likely to be called for in the required testing. Thus, since the reproducibility test would not be intended to provide a statistically valid prediction of expected sampler performance, selection of the site and time of year would be left to the discretion of the tester.

Particle Bounce. Aerosol experts agreed that a procedure for wind-tunnel testing of samplers for particle bounce needed to be developed and evaluated. It was generally agreed that a minimum test would involve 30-μm

*See Part I, Section 3.3.1.

particles. There was less agreement on the need for a solid particle test at the cutpoint. It was generally agreed that the error due to particle bounce should not exceed ±5 percent of the sampling effectiveness obtained for liquid particles of the same size.

Number of Replicate Tests. A tentative value of three replicates at each test point was called for in the draft of the testing procedures. The question arose as to whether three replicates were adequate. Inasmuch as an increase in the number of replicates would increase a whole factorial set of experiments, the consensus seemed to be that it would be preferable to have a specification based on the standard deviation of the three replicates. Additional replicates would be left to the discretion of the tester.

Wall-Loss Tests. In the past, testing of IP samplers has shown that wall losses were a potential problem only for those samplers that further classified the IP fraction. Consequently, the consensus of the group was that wall-loss tests should not be included with the sampling effectiveness tests. However, the group also felt that the entire sampler and not just the inlet should be tested during sampling effectiveness tests so that a candidate sampler that had a wall-loss problem (for liquid particles greater than 5 μm) would probably be detected.

2.2.2 Aerosol Generation

Test Aerosols. Potassium biphthalate may be a good candidate for solid particle bounce tests. Dr. Walter John reported success in generating this aerosol up to 15 μm using the vibrating orifice aerosol generator.

Technical Assistance Document. All attendees were in agreement that aspects of the testing procedures such as particle sizing, slide preparation (liquid particle sizing on oilphobic slides), and particle counting should be contained in a technical assistance document rather than in the testing procedures. Along these lines, Dr. Ben Liu presented results of a study of the flattening factor used to correct the size of the collected liquid particle to the actual physical size. He found that the factor was invariant with particle size and virtually independent of particle composition (within 1.5% for DOP and oleic acid). However, the oilphobic slides had to be carefully prepared according to an established recipe.

Tolerances on Particle Size, Precision of Sizing, Etc. There was general agreement that the precision of particle sizing should be ±0.5 μm, multiplets and satellites should be less than 10 percent, and the generated particle should be within ±5 percent of the nominal testing size.

2.2.3 Wind Tunnel Requirements

Blockage and Turbulence. A requirement that the candidate sampler not block more than 15 percent of the cross-sectional area of the wind tunnel appeared acceptable. A tentative requirement of a 10-percent level of turbulence seemed consistent with the practices at wind-tunnel testing facilities.

Wind Velocity Measurements. Test wind velocity should be within ±10 percent of the speeds indicated in the testing procedures and measured with a precision of 5 percent or better.

Wind Velocity Profile. A reasonable and apparently acceptable specification for the wind velocity profile was that it be uniform over an area one sampler head width by a length equal to five times the sampler head opening.

Choice of Test Wind Speeds. A windspeed test at the U.S. average windspeed should probably be included in the testing procedures, provided that the cost of the wind tunnel capacity would not be excessive.

2.2.4 Artifact Formation: Specification of Filter Media

Dr. Bruce Appel had reported positive artifact (both sulfate and nitrate) formation on glass fiber filters and negative artifact (nitrate) formation on Teflon® filters, but the way this information could or should be used in a specification of the filter media in a Federal reference method for IP was not discussed by the workshop group.

2.2.5 General Considerations: Nomenclature

Considerable debate ensued over the term used to describe the collection of particles by the sampler relative to collection by an isokinetic probe. The term "transmission efficiency" was considered by most to be unsuitable because it connoted a value less than unity, but in cases of enrichment, it would assume a value greater than unity. The term "sampling effectiveness" was preferred by most attendees because it was free from connotations (never having been used in another context); a minority view,

however, held that this asset was actually a liability since anyone outside of the sampler testing clique would have no idea what the term meant. The term "penetration" was preferred by some attendees, but insofar as it is normally defined as one minus the efficiency, it would take on negative values in the case of enrichment. Other terms such as "aspiration efficiency" and "concentration ratio" were clearly considered inferior.

2.3 CONCLUSIONS

2.3.1 Developments in IP Sampling

From the presentations, it was apparent that several sampler inlets had been developed which could meet a performance curve for particle separation at 15 μm at least with liquid particles. This result confirmed the feasibility of basing the reference method on performance specifications.

Based on testing in static chambers, characterization of inlets with solid particles appeared desirable. Wind tunnel tests with solid particles had not been realized although more study was likely to be done in this area.

As in the 1979 IP Workshop, there were difficulties in relating sampler performance in the wind tunnel to sampler performance in ambient air. Data from short-term comparative studies and the IP Network did not seem to bear out the sampler performance predicted on the basis of wind tunnel tests. Resolving this discrepancy was important because sampler testing might need to be modified to include other test conditions or even to eliminate tests that do not produce useful measures of field performance. Clearly, evaluation of the samplers was most readily accomplished under laboratory conditions. Characterizing inlets for particle bounce was considered the next step along with continued intercomparisons in the IP Network.

2.3.2 Filter Media

Concerning specification of the filter media in the IP reference method, the results of Dr. Appel's work do not suggest an obvious course because both glass fiber and Teflon® filters are subject to artifact interferences. (The magnitude of the negative nitrate artifact on Teflon® filters was not considered at the Workshop.) It was not clear that any other choice of filter medium would be free of artifact interferences or

that artifact formation might be a limitation inherent in using filtration to determine aerosol concentration in the atmosphere.

2.3.3 Procedures for Testing IP Samplers

Tentative procedures for testing candidate IP reference method samplers are provided in Appendix C. Recommendations from both the 1979 and 1980 Workshops have been included. Most notable are the acceptance levels for the inlet effectiveness test (Figure 1, Appendix C), tentative tests using solid 30-μm particles (Table C-2, Appendix C), and tests for reproducibility and flow rate stability.

APPENDIX A
ATTENDANCE LIST
OCTOBER 1979 WORKSHOP

APPENDIX A

ATTENDANCE LIST
WORKSHOP ON FEDERAL REFERENCE METHOD (FRM)
FOR INHALABLE PARTICLES (IP)

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APPENDIX B

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NOVEMBER 1980 WORKSHOP

Appendix B

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Workshop on

Federal Reference Method (FRM) for Inhalable Particulates (IP)

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APPENDIX C

TENTATIVE PROCEDURES FOR TESTING PERFORMANCE CHARACTERISTICS OF INHALABLE PARTICLE SAMPLERS*

APPENDIX C

TENTATIVE PROCEDURES FOR TESTING PERFORMANCE CHARACTERISTICS OF INHALABLE PARTICLE SAMPLERS*

1.0 General Provisions

(a) The test procedures given in this subpart shall be used to test the performance of candidate methods against the performance specifications given in Table C-1. A test inhalable particulate sampler representative of the candidate method must exhibit performance better than, or equal to, the specified value for each such specification to satisfy the requirements of this subpart.

(b) For the sampling effectiveness parameter, the test sampler shall be tested by sampling four sizes of liquid particles and one size of solid particles over the size range 5-30 μm, aerodynamic diameter. A wind tunnel having a top speed of at least 24 km/hr (15 mph) shall be used to convey the particles to the sampler. A test point shall consist of testing the sampler performance at a fixed wind velocity, particle size, and particle type. For each of the four sizes of liquid particles, tests shall be conducted at three wind speeds; for the solid particles, tests shall be conducted at two wind speeds. Three replicate measurements shall be made at each combination of wind speed, particle size, and particle type for a grand total of 42 test points (see Table C-2).

(c) For the sampling effectiveness tests, the test point results shall be reported as a performance curve of effectiveness versus particle diameter. For each wind speed, test points from three replicate measurements shall be averaged and plotted against particle diameter. The performance curve shall be constructed by connecting smooth curves between the averaged values at each particle diameter. (At wind speeds of 8 and 24 km/hr, there will be four sizes of liquid particles and one size of solid

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*Recommendations from both the 1979 and 1980 IP Workshops have been incorporated.

TABLE C-1. PERFORMANCE SPECIFICATIONS FOR INHALABLE PARTICULATE SAMPLERS

	Performance parameter	Units	Inhalable particles
1.	Sampling effectiveness	%	Within bounds in Figure 1
2.	Flow rate stability	Lpm	±5% of initial flow over 24 hours
3.	Reproducibility	%	15% precision, Equation in 4.C.5

TABLE C-2. EXPERIMENTAL DESIGN OF SAMPLING EFFECTIVENESS TESTS

Particle size (μm)	Wind speed (km/hr)		
	2	8	24
5	ℓ	ℓ	ℓ
10	ℓ	ℓ	ℓ
15	ℓ	ℓ	ℓ
20	ℓ	ℓ	ℓ
30	-	s	s

ℓ = liquid particle.

s = solid particle.

Number of liquid particle test points (three replicates for each combination of wind speed and particle size): 36.

Number of solid particle tests (three replicates): 6.

Total number of test points: 42.

particles; at a wind speed of 2 km/hr, there will be just four sizes of liquid particles--see Table C-2.) A total of three performance curves of average sampling effectiveness shall be constructed. For the sampling effectiveness test, the performance curve shall be compared to the performance envelope in Figure 1. The test results from each of the three replicate measurements made at a given wind speed and particle size shall be averaged and compared to the specification in Table C-2. A sampling effectiveness performance curve that in any portion lies outside the envelope, or a wall loss value higher than that specified constitutes a failure. These results shall be interpreted as follows:

(1) Zero failures: candidate method passes the performance parameter; and

(2) One or more failures: candidate method fails the performance parameter.

(d) For the flow rate stability test, the flow rate through the test sampler shall be monitored in periods of 24 hours.* If during this period, the sampler flow rate should vary outside the range specified in Table C-1, the test sampler shall have failed the test for that performance parameter.

(e) For the reproducibility test, three identical test samplers, as indicated by the manufacturer, shall be used to sample the inhalable particulate concentration of the open atmosphere for 5 periods of twenty-four hours.† If the reproducibility sould vary outside the range specified in Table C-1, the test sampler shall have failed that performance parameter.

(f) All settings on aerosol generators, wind tunnel speeds, test data, and other documentation obtained from these tests shall be identified, dated, signed by the analyst performing the test, and submitted.

2.0 Test Conditions

(a) Set-up and start-up of the test sampler shall be in strict accordance with the operating instructions specified in the manual. Allow adequate warm-up or stabilization time as indicated in the operating instructions before beginning the tests.

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*The exact number of periods has yet to be prescribed.

†Analogous to CFR, Part 53.33. However, the number of comparison periods and samplers to be tested is tentative.

(b) Once the test sampler has been set up, manual adjustment is permitted only between the testing of the three performance parameters in Table C-1. The submitted records shall show clearly when any manual adjustment or periodic maintenance was made and describe the operations performed.

(c) If the test sampler should malfunction during any of the performance tests, the tests for that parameter shall be repeated. If more than one malfunction occurs, all performance test procedures for all parameters shall be repeated.

(d) Tests for all performance parameters shall be completed on the same test sampler.

3.0 Generation of Test Atmosphere

(a) The test atmosphere shall be generated using a particle generation system and a particle delivery system. The particle generation system shall be capable of producing both solid and liquid particles in the range of 5-30 μm, aerodynamic diameter, with a geometric standard deviation, σ_g, not exceeding 1.1. The particle delivery system shall consist of a wind tunnel having sufficiently large cross-sectional area so that no sampler or sampler head when placed in the wind tunnel shall block more than 15 percent of the area. The particle delivery system shall be capable of achieving uniform wind speeds up to at least 24 km/hr.

(b) The wind speed (air speed) in the wind tunnel shall be determined prior and subsequent to each test run at a particular wind speed. This determination shall be made in accordance with Part 60, Appendix A, Method 2, Determination of Stack Gas Velocity and Volumetric Flow Rate. Under no circumstances shall the average velocity in the wind tunnel differ from the test wind speed in Table C-2 by more than 5 percent.

(c) The accuracy of all flow measurements used to calculate test atmosphere concentrations shall be documented and referenced to a primary standard (such as a spirometer). Any corrections shall be clearly shown. All flow measurements shall be given in actual volume units.

(d) Schematic drawings and other information showing complete procedural details of the test atmosphere generation, verification, and delivery system shall be provided. All pertinent calculations shall be clearly indicated.

4.0 Test Procedures

(a) Sampling effectiveness--1. Technical definition. The ratio of aerosol concentration determined with a test sampler to the "true" aerosol concentration measured by an isokinetic sampler.

2.) Test Procedure

i. Each test point shall be determined by sampling a particulate cloud of monodisperse particles of sizes 5, 10, 15, 20, or 30 μm, aerodynamic diameter, at a known wind speed (see Table C-2). The particulate cloud shall not exhibit greater than 10 percent variation from the mean concentration over an area one sampler head width by a length equal to 5 times the sampler head opening.

ii. For a particular wind speed and particle size (and type), determine the mass concentration using the isokinetic sampling manifold and record as C_{iso}.

iii. Remove manifold and place sampler in wind tunnel. Turn sampler on. Make sure flow rate is properly set. Sample particles for a duration such that the relative error of the measured concentration is less than 5 percent. Relative error is defined as

$$\text{Relative error} = \frac{\text{r.p.}}{x_i} \times 100\%$$

where r.p. denotes the precision of the analytical technique and x_i, the measured concentration, in the same units.

iv. Remove sampler from wind tunnel. Repeat steps ii and iii an additional two times for a total of three replicate measurements at each combination of wind speed and particle size and type (see Table C-2).

v. Determine the amount of material collected on the inhalable particle filter using an appropriate analytical technique (e.g., fluorometry for uranine-tagged aerosol). Calculate indicated mass concentration given by

$$C_i = \frac{\text{amount collected}}{\text{flow rate} \times \text{duration of sampling period}}$$

(Some instruments may read the volume of air sampled, denominator above, directly.) Record the mass concentration as C_{sam}.

vi. Calculate the coefficient of variation for each set of replicate isokinetic sampling measurements. The coefficient of variation, CV, is given by

$$CV = \sqrt{\frac{3}{2}\left(\frac{3\Sigma C^2_{iso,i}}{(\Sigma C_{iso,i})^2} - 1\right)}$$

where $C_{iso,i}$ is the value of a replicate measurement. If the value of CV exceeds 0.10, the set of three replicates must be repeated.

vii. Calculate the average sampling effectiveness for the three replicates as

$$\bar{E}(d_p) = \frac{\sum_{i=1}^{3} \frac{C_{sam,i}}{C_{iso,i}}}{3} \times 100\%$$

where $\bar{E}$, the averaged sampling effectiveness, is a function of particle size; C_{sam} is the concentration measured by the candidate sampler; and C_{iso}, the concentration measured by the isokinetic probe.

viii. Plot average sampling effectiveness against particle diameter for 2, 8, and 24 km/hr wind speeds. The plot shall be on semi-log paper corresponding to Figure 1. (The performance curve for 2 km/hr will contain only four points determined by the liquid particle tests only.)

(b) Flow Rate Stability--1.) Technical definition. Indicated flow rate remains within certain tolerance limits over m sampling periods* of 24 hours.

2.) Test Procedure

i. The test sampler may be adjusted and/or serviced according to the periodic maintenance procedures specified in the manual. The flow rate shall be set to the value prescribed in the manual with a filter in the sampler.†

ii. Connect a flow rate measuring device that provides a recording of flow rate to the test sampler. The sampler and flow rate measuring device shall be connected to the same power line.

iii. The test shall be performed in the open atmosphere. The test sampler shall be run for periods of 24 hours. During the first 10 minutes of each period, a stable trace should be obtained and identified on the strip chart. Should a power failure occur during a 24 hour period, the test shall be repeated. No adjustments of the test sampler shall be made during the test period.

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*As yet undetermined number of sampling periods.

†Whether the flow rate should be at actual or standard conditions has not yet been determined.

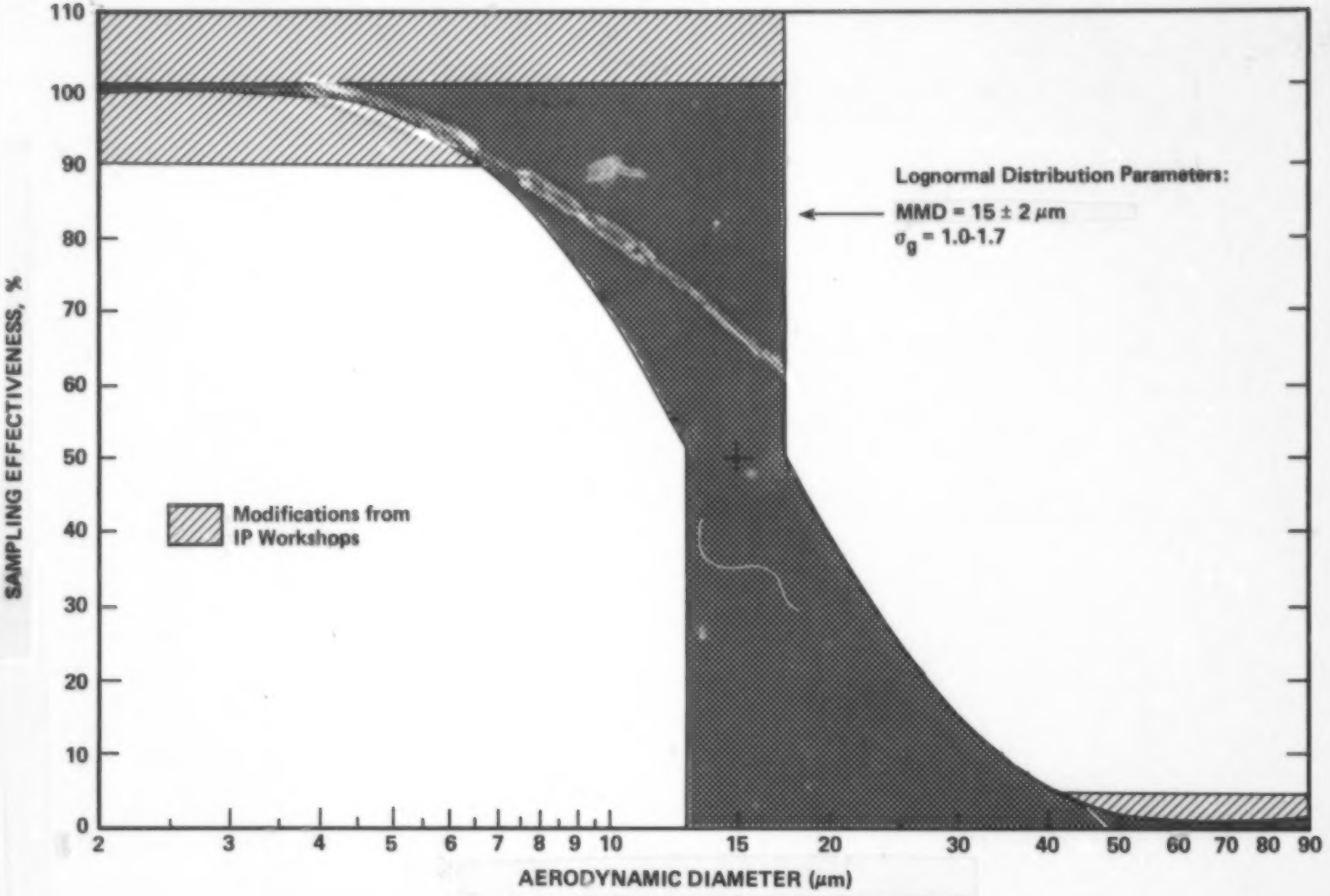

Figure 1. Sampling effectiveness performance envelope.

iv. If the record of the flow rate during any of the periods shows that the flow rate varied by ±5 percent of the initial flow rate, the sampler fails the flow rate stability test.

(c) Reproducibility--1.) Technical definition. Variation of indicated particulate concentrations among identical samplers.

2. The test samplers may be adjusted and/or serviced according to the periodic maintenance procedures specified in the manual. The flow rate shall be set to the value prescribed in the manual.

3. Set up three samplers which are assured by the manufacturer to be identical. Use appropriate filter(s). The samplers shall be connected to the same power line.

4. The test shall be performed in the open atmosphere. The test samplers shall be run for five periods of 24 hours. For each period, calculate the average concentration from the three simultaneously exposed filters and denote it by C_{iave}.

5. Calculate the precision (P) for each of the five periods (in percent) as the maximum minus the minimum divided by the average of the three concentration values, as follows:

$$P_{Ci} = \frac{C_{imax} - C_{imin}}{C_{iave}} \times 100$$

6. If any of the method precision values exceed 15 percent, the sampler fails the reproducibility test.

APPENDIX D

IP SAMPLER INLET INTERCOMPARISON STUDY

Appendix D

IP SAMPLER INLET INTERCOMPARISON STUDY

(Prepared by B. Loo)

An intercomparison of aerosol inlet performance was conducted by EPA in the summer of 1980 in Durham, North Carolina. Four Beckman dichotomous samplers fitted with different inlets were used in side-by-side sampling. Filters from 24-hour samples were analyzed for total mass by beta gauge, and elemental composition by X-ray fluorescence at EPA's Environmental Sciences Research Laboratory. During the course of sampling, the average minimum and maximum wind speeds were 4.3 km/hr and 24 km/hr respectively as measured at the airport, and about 3 inches of rain had fallen.

An examination of the results (Table D-1) revealed that the slopes of one inlet result vs. another were very nearly the same whether one used data for mass or a specific element. Since the X-ray measurement had somewhat higher precision than the beta-gauge measurement, the statistical calcutions presented here were done by choosing the dominant elements Si and S to represent the coarse and fine particle fractions respectively. These partial results are summarized in the following tables. Since the conventional Beckman inlet was the only one used over the entire sampling period, it was represented by the variable 'x'. Variable 'y' was used to represent the other three inlets used, namely Colorado State University (CSU), University of Minnesota-Lawrence Berkeley Laboratory (UM-LBL) and AeroVironment (AV). The number of paired samples considered was denoted by 'N'.

The slope of y vs. x was calculated by a one-parameter least squares fit. Columns 3 and 4 in the table showed two alternative fits corresponding to the cases where data points were weighed by constant fractional error or by constant error. As it turned out, there was no significant difference between the two fits; therefore, the mean slope was calculated. The amount of excess aerosol collected by inlet y over the Beckman inlet is listed in column 5 as a percentage. The error in the slope calculation is listed in the last column.

TABLE D-1. IP SAMPLER INLET INTERCOMPARISON

y	N	$\frac{\Sigma y}{\Sigma x}$	$\frac{\Sigma xy}{\Sigma x^2}$	(Mean Slope)-1 (%)	$\sqrt{\frac{1}{N-1}\left(\frac{\Sigma y^2}{\Sigma x^2} - \frac{(\Sigma xy)^2}{(\Sigma x^2)^2}\right)}$ (%)
			SULFUR (Fine), X = Beckman		
CSU	19	1.030	1.023	2.7	1.3
UM-LBL	21	1.056	1.041	4.9	1.7
AV	9	1.096	1.085	9.1	2.1
			SILICON (Coarse)		
CSU	19	1.014	1.014	1.4	1.6
UM-LBL	21	1.118	1.096	10.7	1.8
AV	9	1.233	1.229	23.1	1.9

It is seen that the error in the slopes was quite small (2%) in all cases, so we may conclude that CSU and Beckman inlets have very close agreement while the UM-LBL and Aerovironment inlets collected significantly more material than the Beckman inlet.

There was no plausible reason why different amounts of fine particles would be collected by different inlets other than that the presumed identical behavior by identical samplers was not true. It is recommended that for intercomparison studies of this type, one needs to alternate the pairing of the inlet to the samplers to average out any unsuspected differences in sampler performance. If we normalize the coarse particle data to the corresponding fine particle data, we would still conclude that the UM-LBL and Aerovironment inlets had collected 5.5% and 12.8% more coarse particles respectively. It should be emphasized that these were only relative measurements since the true ambient aerosol concentration was not known and no inlet had been thoroughly proven to give the best performance.

APPENDIX E

ESTIMATES OF SULFATE AND NITRATE ARTIFACT FORMATION ON GLASS FIBER FILTERS

Appendix E

ESTIMATES OF SULFATE AND NITRATE ARTIFACT FORMATION ON GLASS FIBER FILTERS

(Prepared with the help of B. Appel)

Tables E-1 and E-2 summarize the presentation made by Bruce Appel concerning positive artifact formation on glass fiber filters. For sulfate artifact, maximum sulfate artifact (Table E-1) was estimated on the basis of an observation of about 5 $\mu g/m^3$ artifact sulfate in 24-hr, laboratory exposures of high-volume glass fiber filters (pH = 9.4). Most probable sulfate artifact values (Table E-2) were based on an observation of 3 $\mu g/m^3$ artifact sulfate in 24-hr, high-volume sampling by Appel et al.[1] and Stevens et al.[2] In both cases, artifact sulfate was estimated as the difference between sulfate levels measured on glass fiber filters (several types) and that measured on Teflon® filters during parallel sampling.

For the maximum artifact nitrate estimates (Table E-1), the annual geometric mean nitrate values observed at or near the sites with glass fiber NASN or SCAQMD high-volume samplers were taken as a measure of total inorganic nitrate or TIN (i.e., nitric acid plus particulate nitrate). Nitric acid was then estimated as 50 percent of the TIN values as observed by Appel et al.[3] in Southern California. Most probable artifact nitrate values (Table E-2) were estimated on the basis that nitric acid was 25 percent of TIN values at all sites except Southern California. Appel et al.[4] observed this lower percentage in the San Francisco Bay Area. The nitric acid concentration estimated in this way was then divided by the observed 24-hr mean IP values (Table E-1) to obtain most probable artifact nitrate as a percent of IP. Thus, the most probable total positive artifact estimates with 24-hr, glass fiber, high-volume filters were obtained by merely summing the above percentage figures in Table E-2.

TABLE E-1. ARTIFACT PARTICULATE SULFATE AND NITRATE ON 24-HR HI-VOL GLASS FIBER FILTERS

Sampling size	N	24-Hr mean IP ($\mu g/m^3$)[a]	Data source	Maximum artifact[b] SO_4 (% IP)	Total inorganic NO_3^-[c]		Maximum artifact NO_3^- (% IP)	Benzene-soluble organics	
					$\mu g/m^3$	% IP		$\mu g/m^3$[d]	% IP
New York City	38	107	Stevens et al.	4.6	4.4	4.1	2.1	10.3	9.6
Philadelphia	17	49	Stevens et al.	10	3.5	7.1	3.6	8.1	17
Charleston	102	56	Stevens et al.	8.9	--	--	--	--	--
Portland	17	49	Stevens et al.	10	2.0	4.1	2.1	6.1	12
Chicago	9	43	Wadden et al.	12	4.6	11	6	7.3	15
West Los Angeles	50	46	G. Evans	11	7.0[e]	15	8	--	--
Azusa	17	52	G. Evans	9.6	14.1[e]	27	14	--	--
Ribidoux	35	91	G. Evans	5.5	7.3[e]	8.0	4	--	--

[a] With Teflon® filters.

[b] Based on presumed positive sulfate artifact of 5 $\mu g/m^3$.

[c] NASN hi-vol NO_3^-, annual geometric mean values for 1975.

[d] NASN hi-vol BSO, annual arithmetic mean values for 1966.

[e] SCAQMD, annual geometric mean values for 1976.

TABLE E-2. ESTIMATES OF THE RANGE OF MOST PROBABLE POSITIVE ARTIFACTS WITH 24-HR HI-VOL GLASS FIBER FILTER SAMPLES AS A PERCENTAGE OF INHALABLE PARTICULATE MASS[a, b, c]

Site	Artifact $SO_4^=$ [a] %	Artifact NO_3^- [b] %	Total positive artifact [c] %
All except Southern California	2.8-7.0	1.0-2.7	3.8-9.7
Southern California	3.3-6.5	4.0-14.0	7.3-19.8

[a] Based on assumed positive sulfate artifact of 3 $\mu g/m^3$, i.e.,

$$\% \text{ artifact sulfate} = \frac{3}{IP} \times 100$$

where IP = inhalable particulate mass in Table E-1.

[b] Estimated by the following procedure,

$$\% \text{ artifact nitrate} = \frac{c(TIN)}{IP} \times 100$$

where TIN = total inorganic nitrate (Table E-1),
IP = inhalable particulate mass (Table E-1), and
c = constant, representing fraction of total inorganic nitrate existing as HNO_3.

For Southern California, c = 0.50; and for other areas, c = 0.25.

[c] Sum of ranges in previous columns.

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